

this technique can be found elsewhere [14, 15]. Briefly, a nitrate solution was prepared from the oxides of the corresponding cations. The solution was then atomized using a series of ultrasonic transducers and fed into a vertical tube furnace. The reaction chamber in the furnace was maintained at a temperature range of 600 °C–700 °C, and the residence time of the precursor in the reaction chamber was varied from 0.1 to 60 s. Since the atomized nitrate solution droplets are sufficiently small, they rapidly decompose into metal oxides without loss of lead. The resulting precursor powder is found to be less than 1 μm in size, possesses a narrow particle size range, is composed of many fine grains and has excellent chemical homogeneity from grain to grain. The small particles and grain size of the $\text{Bi}_{1.84}\text{Pb}_{0.34}\text{Sr}_{1.91}\text{Ca}_{2.03}\text{Cu}_{3.07}\text{O}_{10}$ nominal composition precursor allow the oxide phase assemblage to react quickly to form the desired superconducting phase during subsequent heat treatment.

Silver-sheathed PIT tapes were fabricated by the standard PIT process. Precursor powder was packed into an Ag tube of 6.25 mm outer diameter with a wall thickness of 0.75 mm that was open at one end. Powder packing was carried out in a dry box with the aid of a hand press, and the typical packing density is approximately 35–40% of theoretical. The opening of the Ag tube was then capped with an Ag plug, swaged and drawn to roughly 1 mm diameter at 50–100 μm reduction per pass. After drawing, the monofilamentary wire was rolled to approximately 3 mm width and 0.3 mm thickness at 5–10% reduction per pass. The PIT tape was then cut into 2.5 cm long sections for thermomechanical processing.

Three sets of thermomechanical processing experiments were devised in this study. The first set of experiments (set A) was conducted to determine the effect of variation in the number of pressings on the reaction kinetics and J_c of PIT tapes when the total sintering time is kept constant. Since a preliminary test revealed that near-complete Bi-2223 conversion was achieved in roughly 100 h, this time range was chosen as the total sintering time in set A. A listing of the various experimental sequences examined in set A is shown in table 1. The sintering duration is given in hours, and this notation will be used in describing the thermomechanical treatment schedule throughout this paper. A second set of experiments (set B) was performed to determine the optimum duration of an individual sintering step (table 1). The sintering intervals were chosen to be 10, 25 and 50 h, and the thermomechanical treatment was continued for each processing sequence until J_c started to decrease. The last set of experiments (set C) was based on the findings of the previous two sets of experiments, with the aim of determining the effect of changes in sintering duration following the initial sintering step. These intermediate sintering steps are particularly important because they occur after partial Bi-2223 conversion with accompanying retrograde sintering (first sintering interval) and partial alignment of the Bi-2223 crystallites (first pressing) have commenced, and they have been shown to affect the final J_c of fully processed PIT tapes [6–8]. In all the above experimental sequences, sintering was carried out at 825 °C

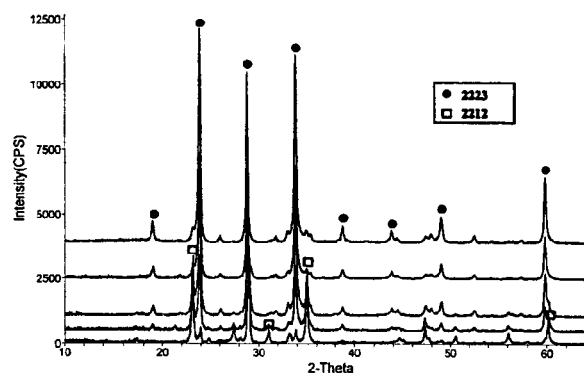


Figure 1. XRD patterns of sequence 1 samples at various stages of thermomechanical treatment.

in 7.5% oxygen–92.5% argon atmosphere with a heating rate of 2 °C min⁻¹ and a cooling rate of 5 °C min⁻¹. Between each sintering step, individual specimens were placed between two steel platens that have been polished with 0.5 μm Al_2O_3 powder and were uniaxially pressed at 1 GPa.

2.2. Sample characterization

The PIT tapes processed by different thermomechanical treatments were characterized in terms of their superconducting and microstructural properties. Critical current density measurements were performed on all the PIT samples at 77 K and self-field using the 1 $\mu\text{V cm}^{-1}$ criterion. The critical current density was determined by dividing the critical current by the cross-sectional area of the superconducting core of the tape. At each stage of the processing cycle a small section of the tape was clipped off and mounted in a mould prepared from resin. The mould was then polished to expose the cross-sectional view of the tape. The area of the superconducting core was measured using an optical microscope equipped with measurable stage movement controls.

Following the J_c measurements, Ag sheaths of selected tapes were chemically etched with an ammonium hydroxide and hydrogen peroxide mixture to expose the HTS core. Evolution of the Bi-2223 phase in the PIT tapes was estimated as the percentage of Bi-2223 (%2223) from x-ray diffraction (XRD) patterns. Peak intensities of the Bi-2212 and Bi-2223 (00l) planes were utilized in estimating %2223 according to the relationship [16]

$$\%2223 = I_{2223} / (I_{2223} + I_{2212}) \quad (1)$$

where I_{2223} is the intensity of the Bi-2223 (0014) peak and I_{2212} is the peak intensity of the Bi-2212 (0012) plane. Microstructural examination of PIT conductors was performed on selected samples using either an optical microscope or a scanning electron microscope equipped with an energy-dispersive spectrometer.